

PREPARATION AND CHARACTERIZATION OF NANO SrTiO_3 CERAMICS

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ABSTRACT

The perovskite structural nanoStrontium Titanate (SrTiO_3) ceramic powders have been synthesized via conventional solid-state reaction method. ST precursor powders were ball milled for nearly 20 h and later undergone calcinations at 1200°C for 9 h and sintered at 1250°C for 3h. The samples were characterized using XRD and FTIR for structural and functional group analysis respectively. The properties such as dielectric constant (ϵ_r), dielectric loss ($\tan \delta$), thermo electric power(S) as well as carrier concentration (η) were studied by making use of HIOKI 3532-50 LCR HiTESTER. The calculated values for high dielectric constants are 110.1 at RT for the frequency 100Hz. Minimum loss (high-Q) at RT is 0.09844.

KEYWORDS: Strontium Titanate (SrTiO_3), Solid State Reaction Method, Dielectric Constant, TEP

INTRODUCTION

Strontium titanate is an oxide of strontium and titanate having ABO_3 perovskite structure with a chemical formula SrTiO_3 . At room temperature it is a centro symmetric paraelectric material with a perovskite structure of density 5.11g/cm^3 . SrTiO_3 ceramics have been extensively used as high promising materials for resonators, tunable capacitors, microwave device components such as filters and phase shifting elements for phased array antennas, and oscillators etc because of their high dielectric constant (K), high permittivity, excellent tunability and low dielectric loss ($\tan\delta$). STO becomes super conducting below 0.35K and was the first insulator and oxide discovered to be super conductive. It is an excellent substrate for epitaxial growth of high temperature super conductors and much oxide based thin films. Its monocrystals can be used as optical windows and high-quality sputter deposition targets.

This material has high dielectric constant about 300[1] and exhibits paraelectricity, non-ferroelectricity at RT. Among metal oxides, crystalline SrTiO_3 is a promising thermo electric material particularly at elevated temperatures. The melting point of SrTiO_3 is 2080°C , making it applicable at high temperatures. Strontium titanate based compounds have applications specifically in tunable electronic devices [2]. In the recent past polycrystalline STO has been discussed in the semiconductor industry as a candidate material for DRAM memories [3]. STO is a quantum paraelectric behavior in which ferroelectric behavior can be induced by appropriate doping through oxygen isotope exchange or under applied stress. Recently STO thin films appeared to be attractive for integrated microelectronic and optoelectronic devices due to growth that is compatible with Silicon technology [4].

PREPARATION OF NANO SrTiO_3 PARTICLES

In order to synthesize the nano strontium titanate ceramic particles SrCO_3 (99.9% purity) and TiO_2 (99.9% purity) were taken as the raw materials. These particles were ball milled for nearly 20h. The resultant nano SrTiO_3 powder particles were calcined at 1200°C for 9h. After wards the pellets were prepared with an addition of very small concentration of binder agent PVA by using hydraulic press with a pressure of 7 to 10 tons and undergone sintering at 1250°C for 3h in order to increase the mechanical strength of pellets. Both flat surfaces of pellets were coated with silver paste.

CHARACTERIZATION OF NANO SrTiO₃

XRD Analysis

X-ray powder diffraction (XRD) is one of the most powerful technique for qualitative and quantitative analysis of crystalline information obtained includes types and nature of crystalline present, structural make-up of phases, degree of crystallinity, amount of amorphous content, micro strain & size and orientation of crystallites. The recorded intensities against 2θ values were plotted for the SrTiO₃. It shows the pattern having peaks related to polycrystalline. Maximum peak was noted at 32.411° with $d=2.74014$.

The lattice parameter is calculated as 3.9125\AA [5] and is in good consistent with the standard value of 3.9541\AA at room temperature. From the (h k l) values i.e. (100), (110), (111), (200), (210), (211), (220), (310) and other results it is confirmed that the structure of the compound is cubic. Furthermore particle size (D), dislocation density (ρ) and elastic strain (E_{strain}) were calculated from the XRD profile. Particle sizes, dislocation densities and elastic strains varied from 28.145nm to 85.568nm, $0.00138(\text{nm})^{-2}$ to $0.001197(\text{nm})^{-2}$ and 0.100-0.245 respectively using the following formulae.

$$D = K\lambda/\beta\cos\theta$$

$$\rho = 1/D^2 \text{ and}$$

$$E_{\text{strain}} = \beta/4\tan\theta$$

The acquired nano size of the strontium titanate powder particles decreases the calcination and sintering temperatures of the sample.

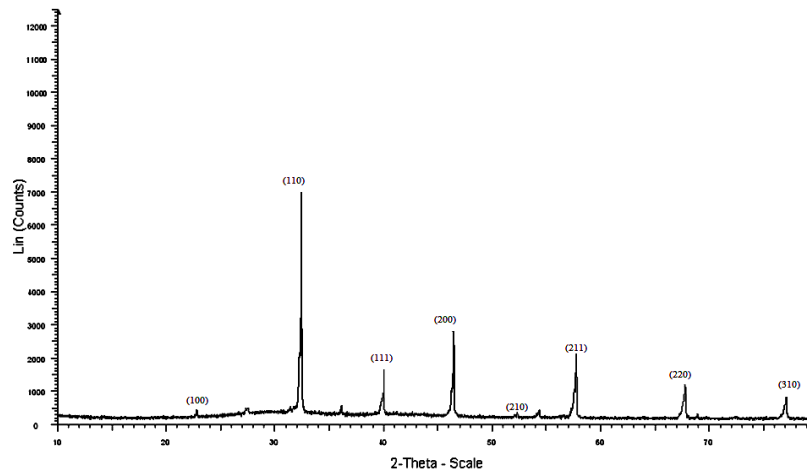


Figure 1: Shows the XRD of Pure SrTiO₃

AC-Conductivity

The following plot illustrates the variation of ac-conductivity (σ_{ac}) with increase of frequency ranging from 100Hz-1MHz for different temperatures of undoped SrTiO₃. The results expressed that the ac-conductivity is increasing as increasing the frequency and temperature of the sample.

At higher frequencies and temperatures the increase in ac-conductivity is very high. This may be due to the strong hopping mechanism. The increase in the graph is linear and therefore this is linear hopping mechanism. In the present investigation the sample showed the maximum σ_{ac} -value of 0.259826 at 1MHz frequency (300°C).

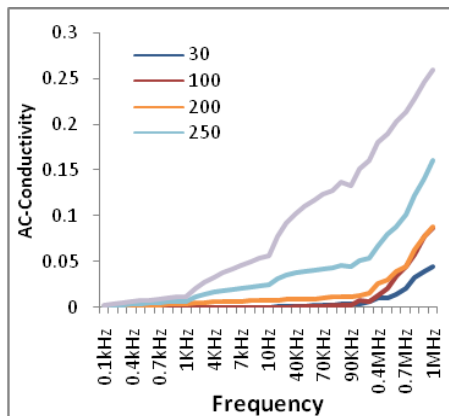


Figure 2: Shows the Frequency vs AC-Conductivity Plot of SrTiO₃

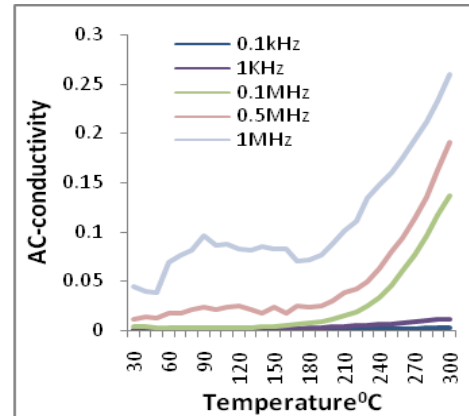


Figure 3: Shows the Temperature vs AC-Conductivity Plot of SrTiO₃

Thermo Electric Properties

The Seebeck coefficient or thermoelectric power and carrier concentration were calculated under thermo electric properties. The calculated values for seebeck coefficient and carrier concentration are 0.0142mv/K and $1.87 \times 10^{22} \text{cm}^{-3}$ $S = \Delta V / \Delta T$ and $\eta = (N/V) \times \exp(-S_e / K_B)$.

Where $N = 10^{22} \text{cm}^{-3}$, $V = 0.6283 \text{cm}^3$, $K_B/e = 86.4 \mu\text{V/K}$

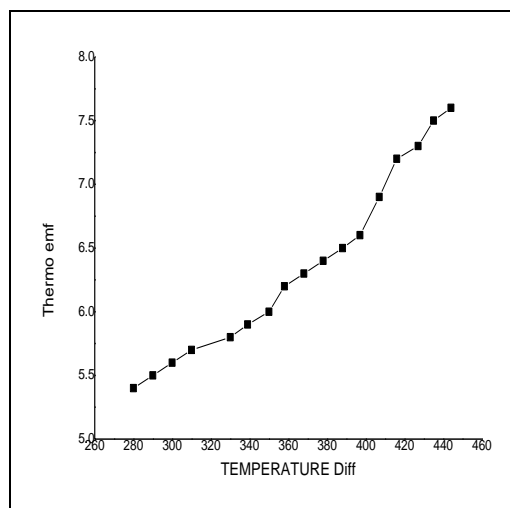


Figure 4: Shows the Thermo emf vs Temperature Difference Plot of SrTiO₃

FTIR Analysis

FTIR analysis helps clients understand materials and products. FTIR testing identifies chemical compounds in polymers, coatings, pharmaceuticals, foods and other products. This also offers both qualitative and quantitative analysis for organic and inorganic samples. In the following FTIR transmittance spectra of undoped SrTiO₃ the peak transmittance 19.47% observed at wave number 1181.4749cm^{-1} and valley transmittance -0.06963% observed at wave number 559.7512cm^{-1} i.e. Transmittance spectrum of undoped SrTiO₃ is observed at near IR-region wavelength of 2.5-25 μm . The functional groups belonging to this region were present in the compound SrTiO₃.

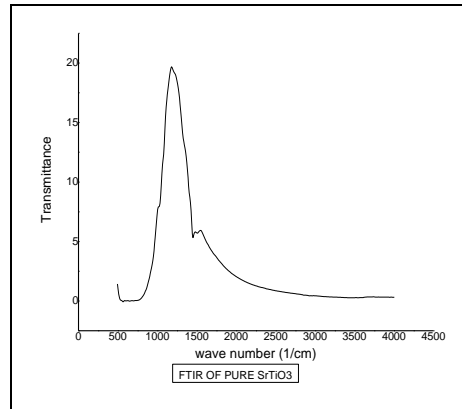
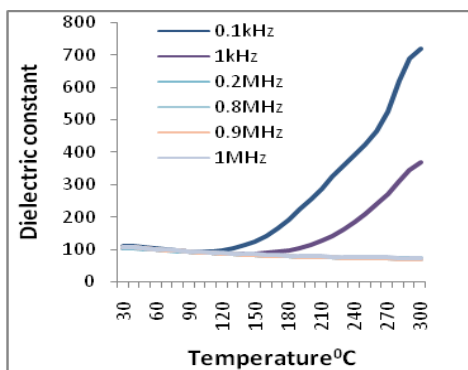
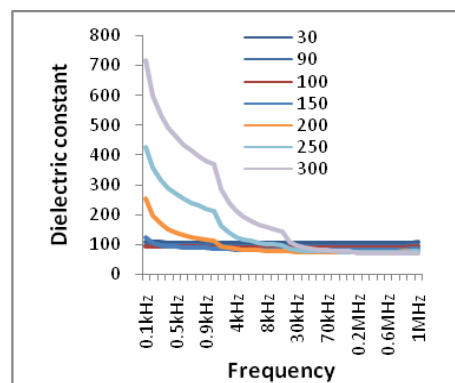
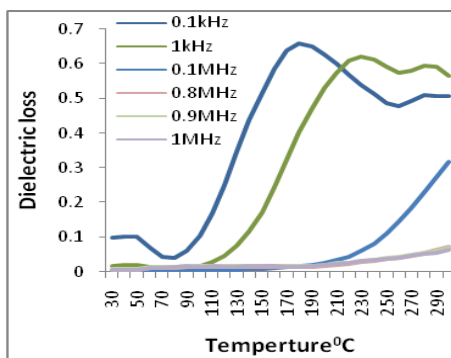
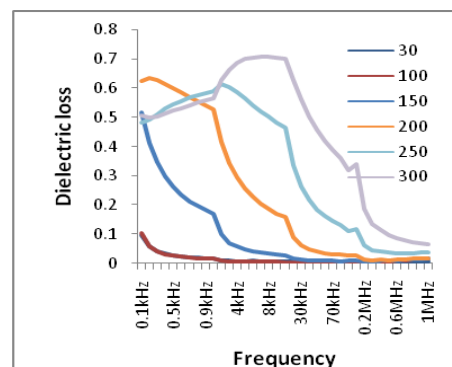


Figure 5

Dielectric Properties

Because of having high dielectric constant [1] and low dielectric loss at room temperature [6, 7] SrTiO_3 can be used as a selective material for capacitors, resonators and filters. The results were accomplished by HIOKI 3532-50 LCR HiTESTER. The results showed that dielectric constant is increasing as the temperature is increasing. At room temperature the maximum dielectric constant obtained is 110.1 while 719.1 at 300°C for the frequency 100Hz. At 1 MHz frequency (at RT) the dielectric constant obtained is 107.6 and approximately as it is at RT(at 100Hz) These results are in good consistent with the standard values. The dielectric loss values measured from 100Hz-1MHz at temperatures from 30°C - 300°C . Minimum loss (high-Q) at 140°C as 0.001. Dielectric constant is measured from equation $K=C/C_0$ or $Cd/\epsilon_0 A$. Where 'd' is the thickness, 'A' is the area of cross section of the pellet and 'C' is the parallel capacitance of the sample. The following plots correspond to the variations of dielectric constant and dielectric loss with respect to the frequency and temperature.

Figure 6: Shows the Dielectric Constant vs Temperature Plot of SrTiO_3 Figure 7: Shows the Frequency vs Dielectric Constant Plot of SrTiO_3 Figure 8: Shows the Dielectric Loss vs Temperature Plot of SrTiO_3 Figure 9: Shows the Frequency vs Dielectric Loss Plot of SrTiO_3

CONCLUSIONS

XRD and SEM were mainly used for the structural and surface morphology studies of SrTiO₃ based ceramic materials. SrTiO₃ attained high dielectric constant and low dielectric loss and therefore beneficial in capacitors needed circuitry systems. Thus SrTiO₃ became selective material for applications in various systems such as microwave device components and tunable capacitors.

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REFERENCES

1. R.C.Neville, B.Hoeneisen and Mead CA, J.Appl.43 (1972)2412.
2. Jaganutsev AK.Sharmen Vo, Astafiev KF, Venkatesh J, Setter N, J electro ceramics (2003), 115.
3. D.E Kotecki, J.D.Baniecki, H.Shen, R.B.Laibowitz and K.L.Saenger IBM J.Res.Develop 43 (1999) 367.
4. Numinous, Proc.IEEE int.Electron devices Mtg (San Francisco, CA, (2008), 929.
5. R.H.Mitchell, A.R.Chakhmouradian, P.M.Woodward, Physics and Chemistry of Minerals 27(2000)583.
6. O.G.Vendik, E.K.Hollmann, A.B.Kozyrev and A.M.Prudan J.Supercond.12 (1999)325.
7. A.K.Tagantsev, V.O.Sherman, K.F.Astafiev, J.Venkatesh and N.Setter J.Electroceram.11(2003)5.

